

Available online at [www.sciencedirect.com](http://www.sciencedirect.com)

Progress in Natural Science: Materials International 21(2011) 491–495

**Progress in  
Natural Science:  
Materials  
International**[www.elsevier.com/locate/pnsc](http://www.elsevier.com/locate/pnsc)

# Effect of cyclic recovery heat treatment on surface recrystallization of a directionally solidified superalloy

Guang XIE<sup>1</sup>, Jian ZHANG<sup>1,2</sup>, Lang-hong LOU<sup>1</sup>

1. Superalloys Division, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China;

2. Shenyang National Laboratory for Materials Science, Institute of Metal Research,  
Chinese Academy of Sciences, Shenyang 110016, China

Received 29 November 2011; Accepted 15 December 2011

**Abstract:** Microstructural evolution and micro-hardness of a directionally solidified Ni-base superalloy subjected to shot-peening during cyclic recovery heat treatment was studied. It was found that  $\gamma'$  dissolved and dislocation annihilation occurred during heating, while  $\gamma'$  re-precipitated when temperature dropped. Due to the formation of a stable dislocation network at  $\gamma/\gamma'$  interface, full recovery and therefore complete elimination of the surface recrystallization are difficult by the present cyclic recovery heat treatment.

**Key words:** recrystallization; superalloy; dislocation; recovery heat treatment

## 1 Introduction

Recrystallization (RX) induced by strain and residual stress in directionally solidified (DS) Ni-base superalloys is a well-known problem in the investment casting industry. It has been reported that the RX is one of the major causes of non-conformance during the processing of single crystal blades [1]. Local RX may reduce the mechanical properties of DS superalloys [2–7], especially under low temperature and high stress conditions [2].

Generally, RX in DS superalloys occurs during heat treatment. The stored energy accumulated during manufacture of the components released by the re-distribution of dislocations at high temperature, which results in the formation of new grain boundaries. In order to reduce or eliminate such detrimental RX, previous work has been concentrated on the following aspects:

1) Introducing particulate phases in order to slow down the migration of dislocation and grain boundary. For example, surface RX occurred frequently in the  $\gamma'$  free zone that formed near the surface due to the selected oxidation of Al and Ti during high temperature heat treatment or mechanical tests [1, 8]. High density of the surface  $\gamma'$  particles can be remained by application of an

overlaying coating which supplied Al and Ti. The surface RX was therefore reduced and the fatigue life of the specimen was greatly enhanced [9–11]. Carbides introduced by carburization may also suppress the surface RX by the pinning effect [12].

2) Removing the surface layer that suffered from plastic deformation prior to heat treatment by chemical milling [13]. The surface RX was significantly reduced after removing the material layer which suffered from severe deformation sufficient to promote the RX upon high temperature exposure.

3) Recovery heat treatment before high temperature solution. In this case, some conflicts existed. WILLIAM et al [14] reported that recovery heat treatment could reduce the depth of surface RX induced by 2%–5% deformation. On the other hand, both long term aging at a temperature 40 °C lower than the final solutionizing temperature, or cyclic recovery annealing failed to prevent recrystallization with 2%–3% deformation in CMSX-11B single crystal alloy [1].

In the present work, the effect of cyclic recovery heat treatment on the surface RX induced by shot-peening in a DS superalloy has been studied in detail. Microstructural evolution was monitored in order to reveal the mechanism of cyclic recovery heat treatment, and explore the method for further optimizing the cyclic

**Foundation item:** Project (2010CB631201) supported by the National Basic Research Program of China; Project (50901079) supported by the National Natural Science Foundation of China

**Corresponding author:** Jian ZHANG; Tel: +86-24-23971196; E-mail: [jianzhang@imr.ac.cn](mailto:jianzhang@imr.ac.cn)

recovery heat treatment parameters.

## 2 Experimental

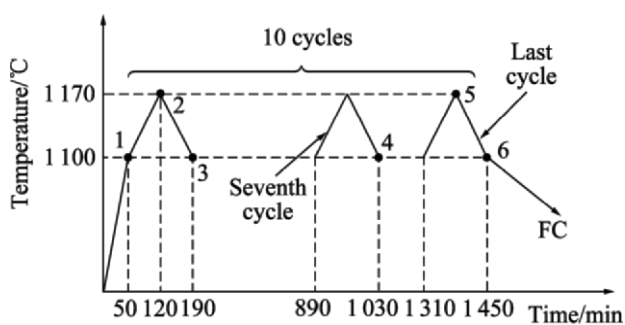
The composition of the DS superalloy studied is 9% Cr, 10% Co, 7% W, 2% Mo, 5% Al, 3.5% Ti, 4% Ta, 0.1% C, 0.01% B and balance Ni (mass fraction). The alloy was directionally solidified into a slab with the size of 220 mm×70 mm×12 mm using a Bridgman furnace. Details of the DS process were reported elsewhere [15].

The slabs were cut into slices with the size of 12 mm×12 mm×3 mm by electron discharge machining (EDM). These slices were ground and shot-peened at a pressure of 0.35 MPa on the plane of 12 mm×12 mm for 1 min. SiO<sub>2</sub> spheres with the radius of 75 μm were used.

The cyclic recovery heat treatment was performed between two temperatures,  $t_1$  and  $t_2$ , both of which are lower than the solutionizing temperature of the alloy. The heating and cooling rates between  $t_1$  and  $t_2$  were about 1 °C/min. All of the samples were cooled to room temperature with furnace and then subjected to solution heat treatment at 1 220 °C for 2 h. In order to observe the microstructural evolution during cyclic recovery heat treatment, some of the recovery annealing cycles were interrupted by quenching the samples into cold water (Fig. 1).

The Knoop micro-hardness of shot-peened samples with and without cyclic recovery heat treatment was examined with the load of 0.098 N for 13 s.

The samples were cut along the cross-section of



**Fig. 1** Schematic diagram of samples used for microstructural observation during cyclic recovery heat treatment

recrystallized surface by EDM. Microstructure of the samples was examined by using optical microscopy (OM) and scanning electron microscopy (SEM). The average depth of RX was obtained based on the measurements every 40 μm along the recrystallized surface from at least 10 photographs for each sample. Dislocation pattern of selected samples was characterized with transmission electron microscope (TEM). Small discs were cut from the shot-peened surface and ground on the opposite side of the surface to 50 μm in thickness. Then, thin foils for TEM were produced with these discs by standard twin-jet polishing techniques. The solution used was 8% perchloric acid in alcohol solution.

## 3 Results

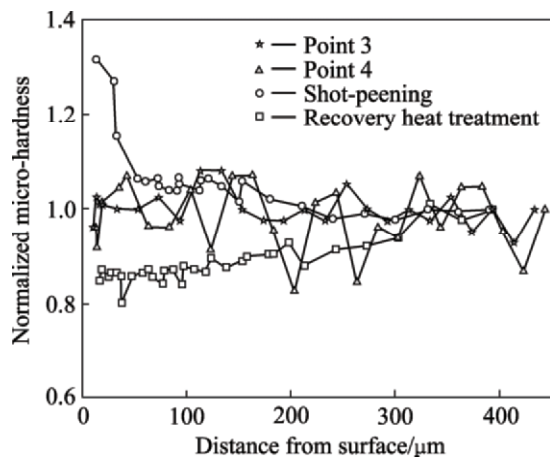
The depth of surface RX in samples with and without cyclic recovery heat treatment is compared in Table 1. Oxidation layer of this alloy heat treated at 1 100 °C for 24 h was about 2 μm [16]. Therefore, the influence of oxidation on data shown in Table 1 was small. In all cases, the average and maximum values of RX depth of the samples after recovery annealing were reduced. High  $t_1$  and  $t_2$  temperature resulted in alleviated surface RX. However, increasing annealing cycles could not significantly reduce the depth of surface RX.

Figure 2 shows the change of Knoop micro-hardness along the cross-section of the samples. Normalized hardness data were shown for the convenience of comparison, since quenched samples generally resulted in a higher micro-hardness. There was obviously a surface layer around 100–150 μm in thickness that was affected by the shot-peening, where high micro-hardness was observed. The most severely deformed area was less than 50 μm where a very high micro-hardness was measured. Cyclic recovery heat treatment reduced this work hardening effect rapidly. Micro-hardness dropped to the level that was similar to that of the un-deformed matrix after only one cycle. However, further annealing showed minor effect on micro-hardness.

Microstructural evolution of shot-peened samples

**Table 1** Comparison of surface RX induced by shot-peening for samples with and without recovery after solution heat treatment

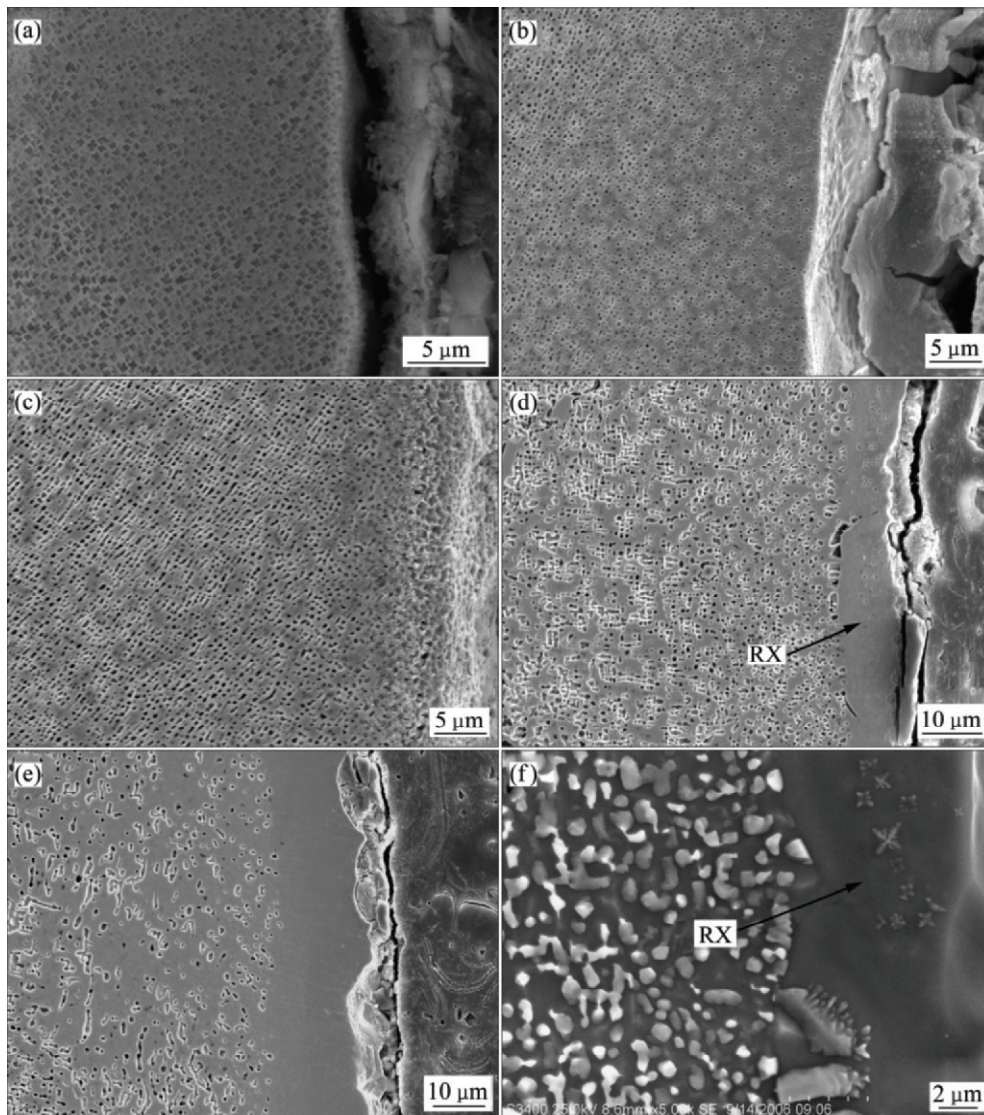
Sample	Number of cycles	$t_1/^\circ\text{C}$	$t_2/^\circ\text{C}$	Heating rate/ $(^\circ\text{C}\cdot\text{min}^{-1})$	Average RX depth/ $\mu\text{m}$	Maximum RX depth/ $\mu\text{m}$
1	10	1 100	1 170	1	17.9±2.1	41±3.2
2	5	1 100	1 170	1	19.1±4.38	44.6±5.21
3	5	950	1 050	1	21.9±3.9	42.3±4.3
Shot-peening	—	—	—	—	26.3±5.14	51.2±6.5



**Fig. 2** Normalized Knoop micro-hardness of shot-peened samples with and without cyclic recovery heat treatment (Normalized hardness is ratio of hardness to average hardness of alloy measured at un-deformed area far away from indentation)

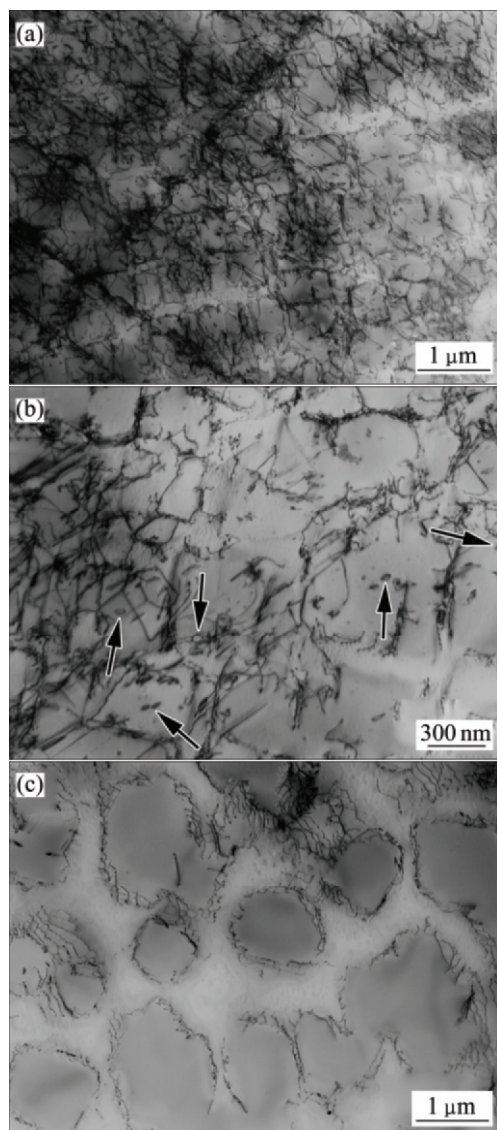
during recovery is shown in Fig. 3. Figures 3(a)–(f) correspond to the interrupted points 1–6 in Fig. 1. By comparing Fig. 3(a) with (c), one can see that  $\gamma'$  near the surface gradually dissolved by increasing the temperature to 1170 °C, and re-precipitated when temperature dropped to 1100 °C. With increasing the number of the cycles,  $\gamma'$  precipitate free zone (PFZ) formed and local RX with an average thickness of 10  $\mu\text{m}$  was occasionally observed (Figs. 3(d)–(f)).

High dislocation density can be seen in the shot-peened samples (Fig. 4(a)). Slip bands and dislocation tangle formed in the  $\gamma$  matrix. A number of dislocation loops generated by Orowan process were also observed (marked by arrows in Fig. 4(b)). On the contrary, very few dislocations can be observed in the  $\gamma$  matrix after cyclic recovery heat treatment, and most of the residual dislocations developed into the dislocation networks at the  $\gamma/\gamma'$  interface, as shown in Fig. 4(c).



**Fig. 3** Microstructural evolution during cyclic recovery heat treatment: (a)–(f) Samples 1–6 in Fig. 1





**Fig. 4** Comparison of dislocation pattern without ((a), (b)) and with (c) cyclic recovery heat treatment after shot-peening

## 4 Discussion

The present results indicated that two  $\{111\}\langle 110 \rangle$  slip systems operated below the surface during shot-peening, which resulted in the work hardening effect and the increase of micro-hardness. During cyclic recovery heat treatment, dislocation loops climbed around the  $\gamma'$  precipitates and shrank until they disappeared at the apices of precipitates [17]. Dissolution of  $\gamma'$  with the increase of temperature decreased the distance of climb and the length of dislocation loops, and therefore accelerated the elimination of dislocation loops. Cross slip of screw dislocations would also occur in the  $\gamma$  channel during heating [18]. Consequently, the annihilation and rearrangement of dislocations occurred, which led to the decrease of the dislocation density, and

therefore the micro-hardness.

It is interesting to note that the recovery occurred rapidly according to Fig. 2. Only one cycle was enough to reduce the micro-hardness to a relatively low level. This means that the dislocation pattern shown in Fig. 4(c) was achieved quickly during annealing, and remained very stable afterwards. Such dislocation configuration is regarded as “equilibrium” structure during high temperature creep of single crystal superalloys [19]. It is therefore believed that the dislocation networks at  $\gamma/\gamma'$  interface shown in Fig. 4(c) are difficult to be eliminated unless the  $\gamma'$  particles dissolve. This explains the reason that increasing the number of the annealing cycles did not significantly reduce the depth of surface RX (Table 1). To further reduce the dislocation density after shot peening, it seems that a higher  $t_2$  is desired.

Many previous works confirmed that RX in DS superalloys (except for the cellular RX) can only occur when  $\gamma'$  precipitates dissolved [1, 20–22], and Fig. 3(d) also clearly demonstrated the pinning effect of the un-dissolved  $\gamma'$ . The present results indicated that when designing the parameters of cyclic recovery heat treatment, it is important that  $t_2$  has to be lower than the solutionizing temperature, while  $\gamma'$  must re-precipitate during temperature dropping from  $t_2$  to  $t_1$ , so that any migration of RX grain boundaries can be retarded. Annealing at a fixed temperature is therefore not an effective way to eliminate the RX.

## 5 Conclusions

Cyclic recovery heat treatment reduced the depth of surface RX induced by shot-peening. The dissolution of  $\gamma'$  precipitates, and the annihilation and rearrangement of dislocations occurred during heating, while  $\gamma'$  particles re-precipitated when temperature dropped from  $t_2$  to  $t_1$ . High dislocation density in the shot-peened sample recovered rapidly, but only partially. Dislocation networks at the  $\gamma/\gamma'$  interface formed during recovery. It is believed that full recovery is difficult to achieve by the present parameters since such dislocation networks are very stable in the directionally solidified Ni base superalloys.

## References

- [1] BÜRGE L, PORTELLA P D, PREUHS J. Recrystallization in single crystals of nickel base superalloy [C]// POLLOCK T M, KISSINGER R D, BOWMAN R R, GREEN K A, MCLEAN M, OLSON S, SCHIRRA J J, ed. Superalloys 2000. New York: TMS, 2000: 229–238.
- [2] ZHENG Y R, RUAN Z C, WANG S C. Recrystallization in surface layer and its effect on stress rupture property for DD22 alloy [J]. Acta Metall Sinica, 1995, 31: 325–329.

- [3] KHAN T, CARON P, NAKAGAWA Y G. Mechanical behavior and processing of DS and single crystal superalloys [J]. *J Met*, 1986, 38: 16–19.
- [4] JO C Y, KIM H M. Effect of recrystallization on microstructural evolution and mechanical properties of single crystal nickel base superalloy CMSX-2 [J]. *Mater Sci Technol*, 2003, 19: 1671–1676.
- [5] SUN C Q, TAO C H, XI N S, ZHANG W F, WU C X. Directionally solidified non-hafnium superalloy and its overload mechanical damage [J]. *Mater Mech Eng*, 2001, 25: 4–7.
- [6] WANG D L. Investigation on recrystallization of Ni-base singlesuperalloy [D]. Shenyang: Institute of Metal Research, 2006: 42. (in Chinese)
- [7] XIE G, WANG L, ZHANG J, LOU L H. Influence of recrystallization on the high temperature properties of a directionally solidified Ni-base superalloy [J]. *Mater Metall Trans A*, 2008, 39A: 206–210.
- [8] XIE G, ZHANG J, LOU L H. Effect of heat treatment atmosphere on surface recrystallization of a directionally solidified Ni-base superalloy [J]. *Scripta Mater*, 2008, 59: 858–861.
- [9] OKAZAKI M, HIURA T, SUZUKI T. Effect of local cellular transformation on fatigue small crack growth in CMSX-4 at high temperature [C]// POLLOCK T M, KISSINGER R D, BOWMAN R R, GREEN K A, MCLEAN M, OLSON S, SCHIRRA J J, ed. *Superalloys 2000*. New York: TMS, 2000: 505–514.
- [10] OKAZAKI M, OHTERA I, HARADA Y. Prevention of fatigue life reduction relating to cellular microstructure in single crystal Ni-based superalloy, CMSX-4, subjected to previous local damage [C]// *Advanced Materials and Processes for Gas Turbines*. TMS, 2003: 63–73.
- [11] OKAZAKI M, OHTERA I, HARADA Y. Damage repair in CMSX-4 alloy without fatigue life reduction penalty [J]. *Metall Mater Trans A*, 2004, 35A: 535–542.
- [12] CORRIGAN J. Single crystal superalloy articles with reduced grain recrystallization, Europe, EP 1038982A1 [P]. 2000–09–02.
- [13] SALKELD R W, FIELD T T, AULT E A. Preparation of single crystal superalloys for post-casting heat treatment, United States, US 5413648 [P]. 1995–05–09.
- [14] WILLIAM J G, JUPITER F. Cycle recovery heat treatment, United States, US 5551999 [P]. 1996–09–03.
- [15] YAN B C, ZHANG J, LOU L H. Effect of boron additions on the microstructure and transverse properties of a directionally solidified superalloy [J]. *Mater Sci Eng A*, 2008, 474: 39–47.
- [16] WANG L. Hot corrosion and high temperature oxidation behaviors of 125 and 1Cr25Ni20Si2 [M]. Shenyang: Institute of Metal Research, 2006: 60. (in Chinese)
- [17] FEDELICH M. A microstructural model for the monotonic and the cyclic mechanical behavior of single crystals of superalloys at high temperatures [J]. *International Journal of Plasticity*, 2002, 18: 1–49.
- [18] POLLOCK T M, ARGON A S. Creep resistance of CMSX-3 nickel-base superalloy single-crystals [J]. *Acta Metall Mater*, 1992, 40: 1–30.
- [19] REED R C. *The superalloys fundamentals and applications* [M]. New York: Cambridge University Press, 2006: 181–185.
- [20] WANG L, XIE G, ZHANG J, LOU L H. On the role of carbides during the recrystallization of a directionally solidified nickel-base superalloy [J]. *Scripta Mater*, 2006, 55: 457–460.
- [21] PORTER A, RALPH B. The recrystallization of nickel-base superalloys [J]. *J Mater Sci*, 1981, 16: 707–713.
- [22] DAHLÉN M, WINBERG L. The influence of g'-precipitation on the recrystallization of a nickel base superalloy [J]. *Acta Metall*, 1980, 48: 41–50.